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(54) IMPROVEMENTS IN PRODUCTION OF AROMATIC ESSENTIAL OILS

(71) We, AGENCE NATIONALE DE VALORISATION DE LA RECHERCHE (ANVAR) a Public Establishment organized under the laws of France, of 13, rue Madeleine Michelis - 92522 NEUILLY-SUR-SEINE CEDEX, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates essentially to a method of high-speed production or expedited manufacture of aromatic or scented essential oils from perfume-generating plants or from component parts thereof, such for instance as seeds, bulbs and flowers as well as apparatus for carrying out the method.

There is already known an apparatus for distilling essential oils from aromatic parts of plants. Such a distillation is performed in accordance with the principle of carrier steam processing.

However the distilling operation is relatively protracted for the following reasons, in particular :

a) the diffusion or desorption of the essential or volatile oils from their secretory cells into the aqueous medium is not a quick phenomenon;

b) the separation of the essential oils from the condensed water after entrainment is based on the principle of continuous decantation. When performing decantation the so-called "florentine" decantors usually employed require moderate liquid velocities;

c) the losses of the most volatile fractions at the condensing system should be reduced as much as possible, and this involves operating the distillation at a reduced speed;

d) finally, if the heat supply into the still is too large, overheating may occur thereby inducing polymerisation or degradation phenomena, as well as the entrainment of solid particles.

The results of such a requirement of conducting the operating step of extracting the essential oils from the aromatic parts of plants in a relatively slow manner or at a relatively low speed is that:

I) the essential oils or perfumes undergo for several hours the action of heat in an aqueous medium. The greater the difficulty of extracting the perfumes, as is the case for instance with seeds or ligneous parts of plants, the longer must be the action of heat thereon;

II) steam distillation and decantation results in recovery only of a liposoluble fraction of the essential oils;

III) incondensable vapours are generally voided to the atmosphere and this may cause nuisance;

IV) the operating cycles are long. In order to minimize in particular the time wasted by loading and unloading, there is a trend to the building of big distilling boilers. Now it is known that when the size of a boiler increases, the ratio of its surface being heated by steam within the double-walled shell or casing, to its useful volume, becomes more and more unfavourable. It is then necessary to increase the working pressure of the steam within the

double-walled shell or casing and accordingly its temperature.

Until now some approaches have been suggested with a view to overcoming the aforesaid drawbacks. It has in particular been tried to inject vapour or steam directly into the still. Such an approach however is likely to destroy the essential oils or perfumes of the plants to be distilled, in particular through degradation on account of the temperature being too high within the still. Moreover this method requires the use of a vapour devoid of extraneous smells likely to interfere with the aromatic essential oils from said plants. Also a relatively substantial fraction of the vapour or steam used for carrying along or entraining the essential oils is liable to condense within the still thereby leading to an excess of condensate being obtained and preventing any reprocessing or recycling of the decanted waters.

Another approach consists in a distilling under pressure. When the pressure prevailing within the still is thus increased, the vapours which evolve therefrom are enriched with entrainable principles, constituents or ingredients (essential oils) because the vapour tension of the essential oils increases faster than the vapour tension of the water when the pressure increases. Consequently the duration of distillation of a given amount of essential oils is reduced. However, as the distillation is effected under pressure, the boiling temperature of the solution is higher and accordingly the aforesaid result is nearly always achieved at the cost of the quality of the product, since effects of polymerisation or thermal degradation are noticed above 100°C. Furthermore, distortions will occur in the composition of the distillate such for instance as a relative enrichment with sesquiterpenes which are undesirable compounds.

The approach according to the invention consists in a method of high-speed production or expedited manufacture, preferably performed batchwise, of aromatic essential oils from perfume-generating plants or from part thereof such for instance as seeds, bulbs, flowers, said method being characterized by the steps of loading said plant material into an enclosure or reactor, comminuting said material in the enclosure or reactor and forming a slurry, substantially simultaneously with this comminution heating said slurry as it is being formed, the temperature being controlled so as to provide the production of vapours with a high content in essential oils, rectifying said vapours in order to obtain vapours with a considerable richness in essential oils and condensing said enriched vapours as a condensate of essential oils which is recovered.

In order to increase the quantity treated in a batch, it is preferable that a part of the material be loaded and comminuted before the remainder is loaded. It is advantageous if, prior to the comminution, the material is loaded into an extraction medium or agent.

Also preferably, once the conversion of the plant material into a slurry has been completed, the heating, carried out during the comminution, is continued under steady stirring (trituration). This heating is advantageously carried out during and after the comminution through heat exchange with a heating fluid or medium.

Thus the method according to the invention enables plant material to be exhausted or depleted of its essential oils in a time three to five times shorter than with the old static process. Therefore, the duration of contact of the volatile oils with the extracting agent and the vapours at the boiling temperature is substantially reduced and their quality is accordingly sharply improved because of less decomposition or polymerisation of said essential oils.

Moreover, this method allows the recovery not only of that decantable fraction of the essential oils which is insoluble in the extracting agent, but also and at the same time a fraction soluble in the extracting agent at a high concentration, due in particular to the aforesaid rectifying step. This fraction which is soluble in the extracting agent may from a quantitative stand-point be as large as the fraction which is not soluble. Preferably the extracting agent is water and with the method it is possible to recover the liposoluble and hydrosoluble fractions of the essential oils. For instance, the hydrosoluble fraction of the essential oils of rosemary is as large as the liposoluble fraction. This is a very significant advantage of the method according to the invention.

Further, the method according to the invention offers the advantage of being applicable to the extraction of the essential oils from very perishable raw materials (such for instance as jasmine) which heretofore could be processed only through extraction with solvents. With the method it is also possible to obtain essential oils from raw materials in which the essential oil content is so low that the essential oils may not be recovered with conventional processes; this is for instance the case with coffee, tea and vanilla.

Another aspect of the invention is an arrangement or apparatus for carrying out the aforesaid method, which is characterized in that it comprises an enclosure or reactor provided with means for comminuting plant material into a slurry, means for regulating the temperature prevailing within said enclosure or reactor, supply means for feeding plant material thereto, means for drawing off the slurry, a distillation column or tower, preferably comprising inclined baffle plates forming trays, which is connected to said

enclosure or reactor, so as to collect and recover the vapours evolving from said slurry, and means for condensing the vapours issuing from the head of said column so as to form at least one condensate of essential oils.

The invention is also directed to the new industrial product consisting of the essential oils and of the aqueous aromatic substances at a high concentration obtained with the aforesaid method.

The accompanying diagrammatic drawings given by way of non-limiting example only illustrate a presently preferred specific embodiment of the invention. In the drawings:

Figure 1 diagrammatically shows an embodiment of an apparatus according to the invention; and

Figure 2 more particularly shows the circuits followed by the extracting agent and by the cooling medium with a view to making substantial savings in the power consumed by said apparatus.

Referring to Figure 1, an apparatus according to the invention essentially comprises at least one enclosure or reactor 1 provided with comminuting means 2 such as a turbo-defibrator or turbo-shredder or turbo-disintegrator or turbo-comminutor or grinder comprising a rotor 3, for instance with discs or blade-knives, and a stator 4 which may or may not be made fast or rigidly connected with the casing of enclosure 1. The stator advantageously comprises blades and in particular swivelling blades. The enclosure 1 also comprises means 5 such as a double envelope or shell, a duct 5a and a feed valve 5b for supplying a heating medium, such for instance as steam, with a view to regulating the temperature within the enclosure 1, means 6 for feeding plant material into the enclosure, and means 7 for drawing off the slurry. The enclosure 1 has means 8 for feeding an extracting agent. Preferably the heating means 5 providing for the regulation of the temperature within the enclosure 1 surrounds the area where the slurry of comminuted plant material is being formed.

The arrangement also comprises a distillation column 9 which is preferably provided with sloping baffle plates forming trays and advantageously has a cross-section of a large size and is connected at its bottom 11 to a co-operating opening 12 of the enclosure 1 so as to collect the vapours evolving from the slurry. Preferably the column 9 is provided with a heat insulation 13. The top 14 of the column 9 communicates with a main device 15 for condensing the vapours flowing out of the column head. This condenser 15 is connected through a pipe-line 16 to a secondary or auxiliary apparatus 17 for condensing the non condensed vapours issuing from the main condenser 15. The apparatus 17 provides a condensation and scrubbing column 20 having a packing 21 and an aftercooler 22, preferably of the reflux type. The condensed volatile essential oils settle in the lower part of the column 18 through the agency of a cooler 19. A drain-cock 23 permits discharge and phase separation through a pipe-line 24 which may communicate with a recycling pump 25 for recirculating the condensation and scrubbing liquid through the agency of a recycle duct 26.

The main condenser 15 is preferably arranged with a small slope or pitch and has its end portion 27, which communicates with the distillation column 9, located on a level higher than the level of the other or opposite end portion 28. The condenser 15 is fitted at the lower part of the end portion 28 with a condensate feedback pipe-line 29 which communicates with a continuous decanting apparatus or settler 30 sealed against air. This settler or decanting device 30 is provided at its bottom part with an outlet 31 which may be connected through a duct 32 and a valve 34 to the top 14 of the column 9. This duct 32 also comprises a draw-off tap or cock 35. The settler 30 includes at its upper part a pipe-line 36 provided with a valve 37 for drawing off the upper phase separated in the settler or decanting device 30.

The settler or decanting device 30 may also be connected at its upper portion through a duct 48 fitted with a valve 49 to the duct 32 downstream of the valve 34.

The condenser 15 comprises a coolant supply pipe-line 38 provided with a control valve 39 for adjusting the feed or supply flow rate and an outflow or output pipe-line 40 for the heated coolant or cooling medium issuing therefrom. Preferably the condenser 15 is of the tubular type but it may exceptionally be a plate condenser or the like in big industrial plants.

Preferably the enclosure 1 is provided with an anti-vortex deflector or like baffle 41 which may be stationary but is preferably adjustable, i.e. swivelling. The temperature-regulating or controlling means 5 comprise a duct 42 for draining or discharging the vapour condensates.

The base portion 43 of the turbo-defibrator or turbo-shredder 2 includes a fluid-tight sealing device for the shaft 44 driving the rotor member 3. The driving of the shaft 44 is preferably provided through a mechanical drive member 45 the speed of which can be varied.

It is also possible to provide a temperature-sensing or detecting means 46 within the enclosure 1 and a temperature-sensing or detecting means 47 within the pipe-line 40, said means preferably consisting of thermocouples. The top section 14 of the column 9 is preferably provided with a filter 50 preventing solid particles from being carried along or entrained from the enclosure 1.

It is also possible to provide a platform 51 for enabling an operator to get access to the apparatus. Means 55 for physically treating or processing the extracting agent may be located downstream of the cock or tap 35 and may then be connected to the duct 32 through a pipe-line 53.

Preferably with reference to Figure 2, the system comprises flow circuits for the extracting agent and the coolant thereby to achieve savings in power. In particular, the circuit for the cooling fluid (which is preferably water) comprises an atmospheric cooling tower 60 providing a storage or supply of coolant which is directed along a closed loop or circuit through the agency of a pump P_1 connected in the pipe-lines 38 and 40. There is also provided a blower or fan system 61.

The flow circuit for the extracting agent comprises a supply tank, jar or like container 62 through which extends the pipe-line 40 preferably forming a tubular coil 40a. The tank 62 communicates through input or feed means 8 provided with a volumetric counter or positive-displacement meter 63. This flow circuit also comprises a supply pipe-line 64 for replenishing or refilling the tank 62 with extracting agent and which is possibly connected through a duct 65 with the draw-off means 7 in order to recover the extracting agent which has issued with the slurry from the container 1 and which, after passing through a heat exchanger 70, has been separated therefrom by a physical treatment device 66. The circuit for the extracting agent moreover comprises on the one hand a pipe-line 67 arranged in a loop about the tank 62 and which is heated in a heat exchanger 68 by the coolant passing through a pipe-line 69 connected to the duct 40. A branch pipe 67a takes part of the extracting agent through the heat exchanger 70 where it is heated by the slurry drawn off the enclosure 1 through the draw-off means 7.

It should be pointed out that the heat exchangers 68 and 70 are of any construction known per se and are for instance of the tubular or plate type. The heat exchange between the flows of hot fluid and cold fluid within the heat exchangers 68 and 70 is effected in counterflow or counter-current relationship so as to provide a methodic and dynamic heat exchange.

The operations of the system described hereinabove is as follows with reference to Figures 1 and 2:

- an amount of extracting agent, which is for instance water and which is predetermined or previously set by the counter or meter 63 is fed from the tank 62 into the casing 1. Then plant material is loaded in bulk into the casing 1 through the feed or input means 6. It may be unnecessary to feed any extracting agent into the casing 1 when the plant material loaded into the casing 1 is likely, upon being comminuted, to release a certain amount of water which then forms the extracting agent. Also when the volume of the plant material put in bulk into the enclosure 1 is too large, part of the material is first put in and comminuted before the rest is loaded. When feeding in an extracting agent such as pure water without any extraneous scent, the volume of extracting agent to be supplied depends upon the treated plant material. This volume is determined through preliminary tests which have given the highest plant material/extracting agent weight ratio consistent with a fluidity of the final slurry adequate for discharge through the draw-off means 7. As a general rule, the plant material-to-extracting agent weight ratio ranges from 0.2 to 1.

After closing off the feed or input means 6 the rotor 3 of the turbo-shredder or defibrator 2 is set into motion thereby effecting a wet comminution of the plant material, thus forming a slurry. This comminution causes the plant material to be divided into fine particles through dilaceration, defibration, disintegration, shredding or breaking of all the parts of the plants loaded in their picked condition. By "parts of the plants" are meant for instance the seeds, the bulbs, the flowers and so on, i.e. all the parts of the plants which are likely to yield aromatic essential oils. The revolving blade or paddle rotor 3 also initiates a vigorous stirring of the slurry thereby further facilitating the exchange between solid and liquid phases and further accelerating the release of essential oils from the plant material or of the volatile oils into the extracting agent.

While carrying out the comminution step, the slurry is simultaneously heated as it is being formed, this heating being at a controlled temperature so as to produce vapours with a high content in essential oils from the plant material by feeding steam through the pipe-line 5a into the double shell 5 of the enclosure or casing 1. The coefficient of heat exchange or transfer at the walls of the double shell 5 is outstanding in view of the turbulence or swirl induced by the rotor 3. This results in the obtainment of a high plant material-to-extracting agent weight ratio and also avoids local overheatings and preferential paths of travel (also

owing to the anti-vortex deflector or baffle 41).

Very quickly, vapours will evolve from the slurry and such vapours are rectified by the column 9 which has a cross-section of adequate size for limiting the flow velocity of the vapours within it and avoiding to the largest extent any entrainment of the solid particles of the plant material. This is particularly important at the lower end of the column at its connection to the enclosure 1; further up the column, the inclined baffle plates 10 are washed by the reflux liquid, compel the vapours to change their direction of flow and inhibit the entrainment of the particles. This also enables high vapour flow rates to be achieved. As an advantage it should be noted that the aforesaid heating accomplished during the comminution step is continued under vigorous stirring action, promoting the obtainment of a good trituration of the slurry once the conversion of the plant material into a slurry has been accomplished. The heating performed during and after the comminution step is advantageously effected through heat exchange with a heating fluid, in particular with steam flowing within the double shell or envelope 5 of the enclosure 1.

The vapours then arrive at the top 14 of the column 9 where the solid materials which they may have carried along are removed therefrom by means of the filter 50 and the vapours then flow through the main condenser 15 where a fraction of the essential oils in admixture with the extracting agent will condense through heat exchange with a cooling medium, which is preferably water flowing in a closed loop or circuit in counter-current relationship through the pipe-lines 38 and 40. The essential oils condensed in admixture with the extracting agent form a condensate. This condensate is subjected to any physical treatment capable of separating all the essential oils from the extracting agent, i.e. capable of separating both the essential oils which are insoluble in the extracting agent and those which are soluble in the extracting agent.

In order to do that, the condensate flows through a pipeline 29 into the settler or decanting device 30 and the essential oils which are insoluble in the extracting agent will separate from the latter while forming two non-miscible phases. If the insoluble essential oils have a specific gravity or density lower than the specific gravity or density of the extracting agent, the phase of the essential oils lies above the phase of the extracting agent and said essential oils are recovered or collected through the duct 36. Preferably the phase of the extracting agent which contains those fractions of the essential oils which are soluble in said agent is drawn off through the cock 35 and subjected, for instance, to an extraction at 55 by means of a solvent or to any other operating step well known in the art allowing the separation from the extracting agent of those fractions of the essential oils which are soluble therein. A predetermined adjustable fraction of the extracting agent is then preferably recycled into the column 9 either through the pipe-line 53 when the fraction is devoid of any essential oils or through the duct 32 whilst it still contains said oils.

Conversely, if the essential oils have a specific gravity or density higher than the specific gravity or density of the extracting agent, the insoluble essential oils are collected or recovered through the cock or tap 35 whereas a fraction of the extracting agent, after separation from the soluble fractions of the essential oils, is recycled to the column 9.

The fraction of vapour which is not condensed within the condenser 15 passes through the duct 16 into the secondary condensing apparatus 17 where it is condensed as a second condensate. With the apparatus 17, it is possible to recover or collect the light fractions of the essential oils which have settled in the lower section 18 of the apparatus 17 and which are recovered or collected through the cock 23.

It should be noted that on the one hand, the proportion in weight of the first condensate (which is collected in the settler or decanting apparatus 30) to the second condensate and on the other hand, the reflux temperature of the extracting agent are adjusted through variation of the flow rate of the cooling fluid of the condenser 15 by means of the valve 39. Through the control or monitoring of the reflux temperature, it is possible to optimize the settling or decanting operation because the light oils would separate better in a hot or warm condition from the extracting agent and a hot or warm reflux would promote the normal working of the column.

As soon as it is observed that no more essential oils are separating from the extracting agent within the settler or decanting apparatus 30, the feed of steam into the double shell 5 is discontinued by closing the valve 5b and the slurry remaining in the enclosure 1 is then discharged through the draw-off means 7 whilst the rotor 3 is still rotating, thereby facilitating the expulsion of the slurry from the enclosure 1.

Preferably, with reference to Figure 2, in order to decrease the consumption of power required for carrying out each operating cycle, after having fed the required volume of extracting agent into the enclosure 1, an equivalent volume of extracting agent is fed into the tank 62. It is thus possible to preheat this extracting agent through heat exchange of the latter with the cooling medium which has flowed through the condenser 15 by means of the heat exchanger 68 and of the pipe-line 40 including the coil 40a in the tank 62. It is also

possible to preheat the extracting agent by recovering the heat from the hot slurry remaining at the end of the cycle through heat exchange in the heat exchanger 70.

Preferably, the slurry remaining at the end of the operating sequence undergoes physical treatment in the physical treatment apparatus 66 in order to separate any non-volatilized essential oils and/or fatty oils contained in the slurry. This physical treatment apparatus 66 may comprise a filtering device, a settling or decanting device, or a device for solvent extraction. The extracting agent separated from the non-volatilized essential oils may then be fed back into the tank 62 through the pipe-line 65, whereas the residual "marc" may be used as a fuel.

It should be pointed out that the extracting agent may be water, a volatile organic solvent, in particular alcohol, or mixtures thereof. The cooling fluid is preferably water, to which an anti-freezing agent may possibly be added for limiting the losses through evaporation. The steam used to heat the enclosure 1 preferably flows in a closed loop or circuit comprising a boiler.

It should further be noted that a part of the mechanical energy consumed during the operation of the rotor 3 of the turbo-defibrator 2 is converted into thermal energy, and this reduces to some extent the supply of heat required for heating the slurry.

All the perfume-generating plants are usable with the method according to the invention. Thus may be cited for instance: jasmine, orange flower or blossom and all flowers with tender petals; rosemary, mint, ginger, mace, allium or garlic, anise, etc... Most of these plants are loaded with an extracting agent such as water with a plant material/extracting agent ratio ranging from 0.2 to 1 and preferably from 0.2 to 0.4; this is the case with allium or garlic and anise. As to those plants which are rich in water (for instance onion) they are comminuted without any addition of extracting agent.

It should be noted that in some cases the slurry is subjected prior to the aforesaid rectifying stage to an enzymatic treatment. This is the case in particular with plants on which an enzymatic action allows the development or even the generation of perfume. Thus, for example, allium and garlic require an enzymatic action to promote the development or the extraction of the essential oils which it is desired to obtain. The comminuting means also accelerates enzymatic action through division and turbulence. The best temperature for performing such an enzymatic treatment is easily obtained with the temperature-regulating or controlling means 5. The enzymatic action is terminated by raising the temperature in order to proceed to the rectifying stage, which simultaneously results in enzymatic inactivation.

As previously mentioned, the invention is applicable to raw materials wherein the content in essential oils is so low that they are not recoverable through the conventional processes: this is for instance the case with coffee, tea and vanilla; and even where there is a lack of separable essential oils, a condensate rich in hydrosoluble or hydrodispersible essential oils may be recovered according to the invention.

To show the superiority of the method according to the invention with respect to the prior art method, the following practical example is given:

- a vessel with a capacity of 1,000 litres is used and 800 litres of water and 300 kg of uncomminuted dry green anise are loaded therein. The comminution means 2 have a rated power of 20 hp. The preheating of the solution is effected in 30 minutes with a corresponding rise of temperature from 20°C to 102°C (the boiling temperature is slightly above 100°C since the pressure is slightly above atmospheric pressure in view of the head or pressure loss in the distilling circuit). The steam pressure in the double shell 5 is 2 bars and the consumption required for the preheating is about 150 kg. The average steam flow rate is about 300 kg/h. For the distilling operation, the heating steam flow rate is about 200 kg/h and the vapourization capacity is about 200 kg/h because said conversion of mechanical energy into thermal energy compensates for the various losses.

Anise oils recovered (as soon as the temperature of the slurry is 102°C):

1st hour	: 4 kg
2nd hour	: 2.5 kg
3rd hour	: 1.5 kg
4th hour	: 1.0 kg
Total amount	: 9.0 kg in 4 hours.

The yield is therefore 3%, the hourly mean distilling speed is 2.25 kg/h whereas the mean speed over the full cycle of 5 hours which comprises the loading, preheating and unloading operating steps is 1.80 kg/h.

The heating steam consumption per kilogram of oil produced is 90 kg whereas the overall steam consumption for the preheating step when the heat recovery system is missing is 105 kg. The cooling water consumption is $3m^3/h$.

According to a conventional method such a recovery of the essential oils from anise would have lasted about 16 hours. Thus the duration of the essential oil recovery operation is four times shorter according to the invention than with the prior art methods thereby resulting in an improvement of the quality of the essential oils and accordingly of the scents and perfumes obtained. Also as previously mentioned, the invention allows both the liposoluble and hydrosoluble fractions of the plants to be obtained which constitutes a very significant advantage.

It should further be noted that the effects of dilaceration during the comminution of the plants may be limited by reducing the peripheral velocity of the rotor 3 or by increasing the spacing between rotor and stator whereby it is possible to avoid the extraction of undesirable substances (for instance of the ligneous parts or pips).

It should be understood that the invention is not at all limited to the embodiment described and shown which has been given by way of example only.

WHAT WE CLAIM IS:

1. A method of high-speed production of aromatic essential oils from perfume-generating plants or from parts of said plants, such as for instance seeds, bulbs, flowers, characterized by the steps of loading said plant material into an enclosure or reactor, comminuting said material in the enclosure or reactor and forming a slurry, substantially simultaneously with this comminution heating said slurry as it is being formed, the temperature being controlled so as to provide the production of vapours with a high content in essential oils, rectifying said vapours in order to obtain vapours with a considerable richness in essential oils and condensing said enriched vapours as a condensate of essential oils which is recovered.

2. A method according to claim 1, the said method being performed batchwise.

3. A method according to claim 2, wherein part of the material is loaded and comminuted before the remainder is loaded.

4. A method according to any one of the preceding claims, wherein the plant material is comminuted in an extracting agent.

5. A method according to claim 4, wherein the plant material/extracting agent weight ratio lies between 0.2 and 1.

6. A method according to claim 4 or claim 5, wherein the plant material is loaded into preheated extracting agent in the enclosure or reactor.

7. A method according to any one of claims 4 to 6, wherein the extracting agent is selected from water, a volatile organic solvent and mixtures thereof.

8. A method according to any one of claims 4 to 7, wherein a cooling medium for the condensation is recycled for heating the extracting agent.

9. A method according to any one of claims 4 to 8, wherein at least part of the condensate is subjected to treatment likely to separate all the essential oils from the extracting agent, said separated essential oils comprising the essential oils soluble in the extracting agent.

10. A method according to claim 9 wherein an adjustable predetermined fraction of the separated extracting agent is recycled to the rectifying step.

11. A method according to any one of the preceding claims, wherein the heating is continued under vigorous stirring after the conversion of the plant material into a slurry has been effected.

12. A method according to any one of the preceding claims, wherein the heating is effected through heat exchange with a heating fluid.

13. A method according to any one of the preceding claims, wherein two condensation steps are performed, viz. a first partial condensation of the enriched essential oil vapours, thereby obtaining a first condensate and a second, condensation of the non-condensed essential oil vapours following said first partial condensation thereby obtaining a second condensate, said condensations being carried out through heat exchange with a cooling medium.

14. A method according to claim 13 wherein on the one hand, the proportion of the first condensate to the second condensate and on the other hand, the reflux temperature of the extracting agent are controlled or adjusted through variation of the flow rate of the cooling fluid inducing the partial condensation.

15. A method according to any one of the preceding claims, wherein the slurry remaining at the end of the cycle is subjected to a physical treatment to separate the

non-volatilized essential oils and/or fatty oils contained in the slurry.

16. A method according to any one of the preceding claims, wherein the slurry is subjected to an enzymatic treatment before rectification, the desired temperature for performing this treatment being obtained by means of a temperature regulator, the enzymatic action being terminated by raising the temperature for passing to the rectifying stage.

17. Apparatus for carrying out the method according to claim 1, characterized in that it comprises an enclosure or reactor provided with means for comminuting plant material into a slurry, means for regulating the temperature prevailing within said enclosure or reactor, means for feeding plant material thereto, means for drawing off said slurry, a distillation column connected to said enclosure or reactor so as to collect and recover the vapours evolving from said slurry and means for condensing the vapours issuing from the head of said column so as to form at least one condensate of essential oils.

18. Apparatus according to claim 17 wherein the distillation column comprises inclined baffle plates forming trays.

19. Apparatus according to claim 17 or claim 18, wherein said comminuting means comprise a turbo-defibrator or turbo-disintegrator including a rotor and a stator.

20. Apparatus according to claim 19 wherein said rotor is a rotor provided with discs.

21. Apparatus according to claim 19, wherein said rotor is a rotor provided with blade-knives.

22. Apparatus according to any one of claims 19 to 21, wherein said stator comprises blades or paddles, preferably swivelling blades.

23. Apparatus according to any one of claims 17 to 22, wherein said means for regulating the temperature in said enclosure or reactor surround the area where the slurry of the comminuted plants is being formed.

24. Apparatus according to claim 23, wherein said enclosure or reactor comprises a double-walled vessel and is fitted with a pipe-line and a valve for supplying a heating fluid such for instance as steam.

25. Apparatus according to any one of claims 17 to 24, wherein said enclosure or reactor is fitted with an antivortex deflector.

26. Apparatus according to any one of claims 17 to 25, including at least one device for physically treating the condensate and in particular at least one settler or decanting device and/or at least one device for extraction by means of a solvent.

27. Apparatus according to any one of claims 17 to 26, wherein means are provided for feeding extracting agent to the enclosure or reactor.

28. Apparatus according to any one of claims 17 to 27 comprising a main condenser for the partial condensation of the vapours through heat exchange with a cooling fluid and a secondary condenser for the final condensation of the vapours as well as a duct for recycling the extracting agent to said distillation column.

29. Apparatus according to claim 28 comprising a heat exchanger, where the extracting agent is heated by the cooling fluid which has flowed through said main condenser, and a control valve for adjusting the flow rate of said cooling fluid, said valve being connected into the pipe-line feeding the cooling fluid to the main condenser.

30. Apparatus according to any one of claims 17 to 29, wherein the connection of said column to said enclosure or reactor has a cross-section of adequate size to limit the velocity of the vapours passing therethrough and to avoid excessive entrainment of solid particles of the plant material from the slurry.

31. Essential oils and aqueous aromatic substances at high concentration, obtained by the method according to any one of claims 1 to 16.

32. A method according to claim 1 substantially as described herein.

33. Apparatus substantially as described herein with reference to and as shown in the accompanying drawings.

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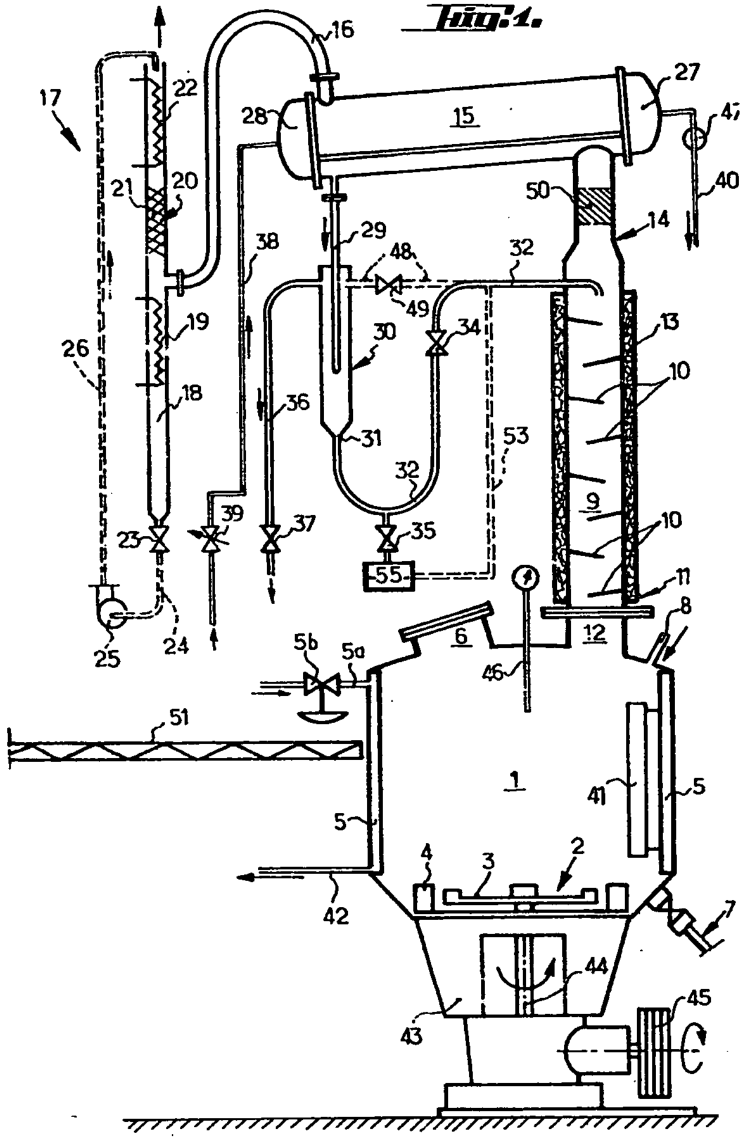
1600942 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 1

Fig. 1.



1600942 COMPLETE SPECIFICATION

2 SHEETS *This drawing is a reproduction of the Original on a reduced scale*

Sheet 2

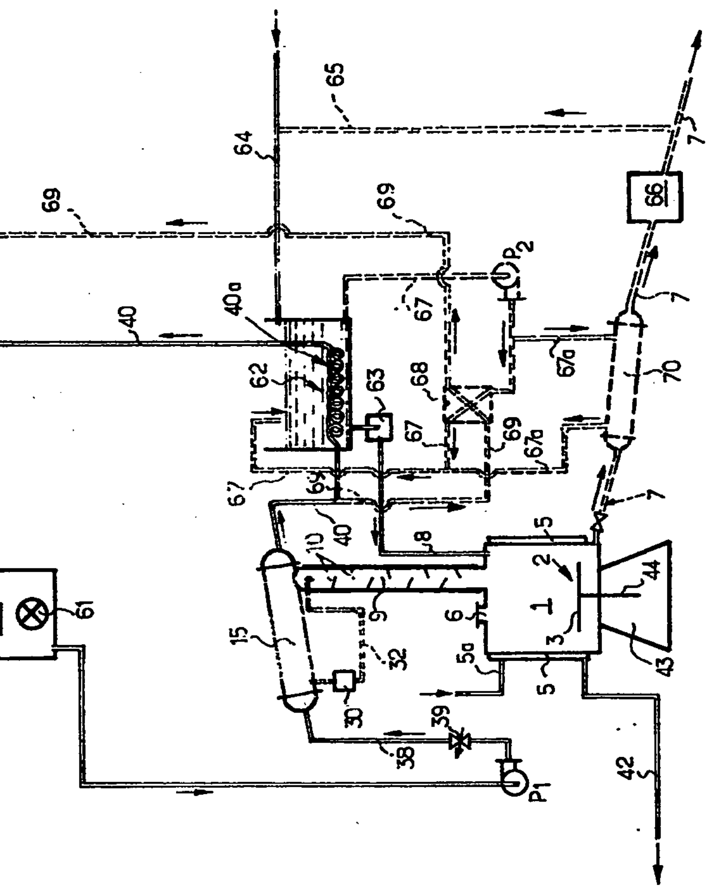


Fig. 3.